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## CERTIFICATION UNDER 37 CFR 1.10

Thereby certify that this Transmittal Letter and the papers indicated as being transmitted therewith are being deposited with the United States Postal Service on this date shown below in an envelope as "Express Mail Post Office to Addressee" under the below indicated Mailing Label Number, addressed to: Box Patent Application, Assistant Commissioner for Patents, U.S. Patent & Trademark Office, Washington, D.C. 20231.

Mailing Label No.: EL592218610USDeposit Date: September 21, 2001  
Name: Amanda L. AmatoATTORNEY'S DOCKET No. CULLP0161US

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE  
(DO/EO/US)**

In re national phase of:

Applicant(s): Leigh Albert Sullivan, David Murray McConchie  
& Richard Bush  
International Application No.: PCT/AU00/00224  
International Filing Date: 22 March 2000  
Priority Date Claimed: 22 March 1999  
Title of Invention: APPARATUS FOR ANALYZING REDUCED  
INORGANIC SULPHUR

**TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED  
OFFICE (DO/EO/US) CONCERNING ENTRY INTO U.S. NATIONAL  
PHASE UNDER 35 U.S.C. 371**

Box Patent Application  
Assistant Commissioner for Patents  
U.S. Patent & Trademark Office  
Washington D.C. 20231

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information under 35 U.S.C. 371:

1. This express request to immediately begin national examination procedures (35 U.S.C. 371(f)).
2. The U.S. National Fee (35 U.S.C. 371(c)(1)) and other fees (37 CFR 1.492) as indicated below.

3. A copy of the International application (35 U.S.C. 371(c)(2)):
- a. ☒ is transmitted herewith  
(International Publication No. WO 00/57174 ).
  - b. ☐ is not required, as the application was filed with the United States Receiving Office.
  - c. ☐ has been transmitted by the International Bureau. A copy of Form PCT/1B/308 is enclosed.
4. ☐ A translation of the International application into the English language (35 U.S.C. 371(c)(2)) is transmitted herewith.
5. Amendments to the claims of the International application under PCT Article 19 (35 U.S.C. 371(c)(3)):
- a. ☐ are transmitted herewith.
  - b. ☐ have been transmitted by the International Bureau.
6. ☐ A translation of the amendments to the claims under PCT Article 19 (38 U.S.C. 371(c)(3)) is transmitted herewith.
7. A copy of the international examination report (PCT/IPEA/409)
- a. ☐ is transmitted herewith.
  - b. ☐ is not required as the United States Patent and Trademark Office was the IPEA.
8. Annex(es) to the international preliminary examination report
- a. ☐ is/are transmitted herewith.
  - b. ☐ is not required as the United States Patent and Trademark Office was the IPEA.
9. ☐ A translation of the annexes to the international preliminary examination report is transmitted herewith.
10. ☒ An oath or declaration of the inventor (35 U.S.C. 371(c)(4)) complying with 35 U.S.C. 115 is submitted herewith.

11. An International Search Report (PCT/ISA/210)
- a. ☒ is transmitted herewith.
  - b. ☐ has been transmitted by the International Bureau.
  - c. ☐ is not required, as the application was searched by the United States International Searching Authority.
12. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98 is transmitted herewith, along with Form PTO-1449 and copies of citations listed.
13. ☒ An assignment document is transmitted herewith for recording, along with a separate cover sheet.
14. ☐ A preliminary amendment is enclosed.
15. ☒ Applicant claims small entity status.
16. ☐ Other:

Basic National Fee					Fee
IPEA - US					\$690.00
ISA - US					\$710.00
PTO not ISA or IPEA					\$1,000.00
Claims meet PCT Art. 33(1)-(4) - IPEA - US					\$100.00
Filing with EPO or JPO search report					\$860.00
Enter appropriate basic fee →					\$1,000.00
Claims*	Number filed		Number extra	Rate	
Total claims	26	-20	6	\$18.00	\$108.00
Independent claims	1	-3	0	\$78.00	\$0.00
Multiple dependent claims (if applicable)				\$260.00	
Total of above					\$1,108.00
Small entity statement enclosed, 1 if Yes, 0 if No →				1	\$554.00
Total national fee					\$554.00
Fee for recording enclosed assignment				\$40.00	\$40.00
Total fees enclosed					\$594.00

\*After any attached preliminary amendment reducing the number of claims and/or deleting multiple dependencies.

☒ [ X ] A check in the amount of \$ 594.00 to cover the above fees is enclosed.

☐ [ ] Please charge our Deposit Account No. 18-0988 in the amount of \$           . A duplicate copy of this sheet is enclosed.

WARNING: TO AVOID ABANDONMENT OF THE APPLICATION THE BASIC NATIONAL FEE MUST BE PAID WITHIN THE 20/30 MONTH TIME LIMIT.

16. The Commissioner is hereby authorized to charge the following additional fees that may be required by this paper and during the entire pendency of this application to our Deposit Account No. 18-0988 under Attorney Docket No. CULLP0161US:

a. ☒ 37 CFR 1.492(a)(1), (2), (3), (4) and (5) (filing fees)

WARNING: BECAUSE FAILURE TO PAY THE NATIONAL FEE WITHIN 30 MONTHS WITHOUT EXTENSION (37 CFR S 1.495(B)(2)) RESULTS IN ABANDONMENT OF THE APPLICATION, IT WOULD BE BEST TO ALWAYS CHECK THE ABOVE BOX.

b. ☒ 37 CFR 1.492(b), (c) and (d) (presentation of extra claims)

NOTE: Because additional fees for excess or multiple dependent claims not paid on filing or on later presentation must only be paid or these claims cancelled by amendment prior to the expiration of the time period set for response by the PTO in any notice of fee deficiency (37 CFR 1.492(d)), it might be best not to authorize the PTO to charge additional claim fees, except possibly when dealing with amendments after final action.

Respectfully submitted,



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**APPARATUS FOR ANALYSING REDUCED INORGANIC SULFUR**  
**FIELD OF THE INVENTION**

The present invention relates to an apparatus for the quantitative analysis of reduced inorganic sulfur.

5

**BACKGROUND ART**

Sulfur is present in the environment in several different forms which may be generally classified as organic and inorganic sulfur. The presence of each form of sulfur has important environmental implications. For example, combustion of both inorganic and organic sulfur produces sulfur dioxide, a greenhouse gas and source of acid rain. Another significant environmental problem is the acid generating potential of a class of inorganic sulfur materials known as reduced inorganic sulfur. These compounds produce acid when materials containing them are mined, excavated or drained, and exposed to atmospheric oxygen.

15

The term "reduced inorganic sulfur" refers to sulfur in a form that can undergo oxidation and includes mineral disulfides (e.g. pyrite and chalcopyrite), monosulfides (e.g. sphalerite, galena and covellite), polysulphides (e.g. pyrrhotite and bornite), non-stoichiometric metal sulfides (e.g. greigite), other sulphides such as chalcocite, sulfites (salts of sulfurous acids) and elemental sulfur. In the present specification and claims the term "reduced inorganic sulfur" will be understood to include any inorganic sulfur compound that can be oxidised.

20

Further types of naturally occurring inorganic sulfur compounds are the sulfate minerals, such as gypsum, which include sulfur in the oxidized sulfate form. These materials are generally not a source of acid in the environment.

25

When sulfide bearing material is mined or excavated, oxidation of the reduced inorganic sulfur may occur. The result is acid sulfate soil and acid mine drainage. Acid mine drainage refers to acid water produced by the oxidation of minerals such as pyrite in the presence of water and is one of the major environmental problems facing the mining industry. The oxidation of reduced inorganic sulfur in acid sulfate soils is a global environmental problem affecting more than 12 million hectares of agricultural land worldwide,

30

degrading aquatic habitats and corroding concrete and steel infrastructure. It is therefore important for environmental management to be able to measure the amount of reduced inorganic sulfur in a wide range of materials including sediments, soils, mine spoil, sludge, petroleum, mineral ores, fossil fuels and water.

There are several known methods for measuring the sulfur content of a material. Combustion followed by measurement of evolved sulfur oxides is used to measure the total sulfur content (i.e. the combined inorganic and organic sulfur). Such methods are typically used to measure the sulfur content of coal. Combustion of coal is a major source of sulfur dioxide pollution. However, the combustion method does not distinguish between organic sulfur, reduced inorganic sulfur or mineral sulfates and accordingly cannot be used to accurately quantify the reduced inorganic sulfur in a sample.

The most widely used method for assaying reduced inorganic sulfur is by peroxide oxidation in which the sulfur is oxidised to sulfate. The amount of sulfate liberated by peroxide oxidation is then analysed by conventional wet chemical quantitative analysis. Although this method is the accepted procedure, the present inventors have surprisingly and unexpectedly observed that the method is subject to serious interferences from organic sulfur and sulfate minerals such as gypsum. This interference is particularly important when sediments having low reduced inorganic sulfur are measured. An erroneous estimate of the reduced inorganic sulfur content may lead to the recommendation of costly and/or inappropriate and environmentally damaging management practices.

Other methods for analysing reduced inorganic sulfur, but which are considered to be less accurate than the peroxide oxidation method, include:

- (a) measuring the total sulfur and soluble sulfur content and estimating the reduced inorganic sulfur content from the difference between the two values. A disadvantage of such differential measurement is that the errors are cumulative.

- (b) microscopic examination of a sample, and
- (c) indirect measurement by estimating the amount of pyritic iron ( $\text{FeS}_2$ ) in a sample. However non-pyritic forms of reduced inorganic sulfur are not measured.

5           It is therefore an object of the present invention to provide a method and apparatus for measuring the reduced inorganic sulfur content of a sample selectively and accurately.

### DISCLOSURE OF THE INVENTION

10           According to a first broad form of the invention there is provided an apparatus for measuring the reduced inorganic sulfur content of a sample, the apparatus having a reaction chamber, means for introducing a reducing agent that can selectively convert the reduced inorganic sulfur of a sample into the reaction chamber to hydrogen sulfide, means for measuring the amount of hydrogen sulfide evolved by reaction of the reducing agent with the  
15           sample and a detector for detecting when evolution of the hydrogen sulfide has reduced or ceased.

20           The apparatus of the present invention has a reaction chamber for allowing a sample to be tested to be digested by a reducing agent that selectively converts the reduced inorganic sulfur component of a sample to  $\text{H}_2\text{S}$ . The reducing agent should not react with organic sulfur or sulfate materials to produce  $\text{H}_2\text{S}$ . Preferred reducing agents include chromous salts, stannous salts and mercurous salts.

25           An especially preferred reducing agent is acidified chromous chloride. Acidified chromous chloride solutions are typically prepared by passing acidified chromic chloride through a column containing zinc, pre-amalgamated in mercuric nitrate. This process is difficult, slow and requires specialised equipment to minimise atmospheric oxidation of the acidified  $\text{CrCl}_2$ . Also, acidified  $\text{CrCl}_2$  is unstable and can only be stored for a few days. Thus, it is preferred that chromous chloride is generated in situ in the reaction  
30           chamber. Typically, chromium, concentrated  $\text{HCl}$ , ethanol and distilled water are introduced into the reaction vessel. The chromium is typically in the form of a powder, but may also be added as a pellet or slurry.



The reduction reaction is typically carried out in an inert atmosphere such as nitrogen or argon, but can for short periods of time be conducted in air, such as up to about 20 minutes. Generally, the reaction is carried out at elevated temperatures with or without agitation under reflux conditions. The condenser used in the reflux process may be cooled by any of the known methods including a continuous flow of water or a refrigeration unit. In a particularly preferred embodiment of the invention, the apparatus includes a condenser that is cooled by means of water that is recirculated through a refrigeration unit. This arrangement allows for the apparatus to be in the form of a portable unit that is suitable for use in the field.

The apparatus of the invention is typically automated and controlled by a central processor which can control some, or essentially all, of the functions of the apparatus. This allows the apparatus to be operated by non-skilled personnel.

Typically, the amounts of reagents added to the reaction chamber are automatically supplied to the reaction chamber in pre-determined quantities in a pre-determined order. Generally, the liquid ingredients are added using a peristaltic pump.

In a further broad form of the present invention there is provided an automated apparatus for measuring the reduced inorganic sulfur content of a sample, the apparatus having a reaction chamber, means for heating the chamber, a condenser, means for introducing a reducing agent into the reaction chamber which converts reduced inorganic sulfur to hydrogen sulfide and measuring means for measuring the amount of hydrogen sulfide evolved.

Typically, the apparatus includes a refrigeration unit for cooling water for the condenser.

The reduced inorganic sulfur composition of the sample may be calculated from the amount of  $H_2S$  evolved by reaction with the reductant. The  $H_2S$  evolution may be measured by any of the known methods of measuring  $H_2S$ . Such methods include colourimetric, turbidimetric and gravimetric methods. Particularly preferred methods include electrochemical, spectroscopic or chromatographic techniques such as mass spectroscopy,

gas chromatography, UV or IR spectroscopy. A particularly preferred measurement means is an electrochemical gas analyser. Generally, the gas which provides the atmosphere in the reaction chamber also serves as a carrier gas to carry the evolved  $\text{H}_2\text{S}$  to the measurement means.

5           The amount of  $\text{H}_2\text{S}$  evolved may also be measured indirectly. For example,  $\text{H}_2\text{S}$  may be oxidised to sulfate or sulfur dioxide. These oxidation products may then be measured by suitable methods including turbidimetric or gravimetric, chromatographic or spectroscopic methods.

10           The apparatus of the present invention typically includes a means for detecting when the evolution of  $\text{H}_2\text{S}$  has decreased to a pre-determined rate. Typically, this is when  $\text{H}_2\text{S}$  evolution has substantially ceased. Cessation of  $\text{H}_2\text{S}$  evolution indicates that the reduction reaction has been completed. When completed, a fresh sample may be analysed. With conventional wet chemical procedures, a sample is allowed to react for a pre-determined maximum length of time. The present inventors have observed  
15           that the reduction reaction can often take much less time than has traditionally been allowed. Thus, by being able to monitor when the reaction has ceased, sample throughput may be optimised. The apparatus may include an alarm or other signalling device to alert an operator that the reaction has finished.  
20           Alternatively and/or in addition to, the apparatus may have means for automatically disengaging any heating of the reaction chamber and deactivate the condenser on completion of the reduction reaction.

25           The means for detecting when evolution of  $\text{H}_2\text{S}$  has ceased may be in addition to, or part of, the  $\text{H}_2\text{S}$  measuring means. For example a gas sensor or other detection means may be associated with the reaction chamber to detect the  $\text{H}_2\text{S}$  therein. Gas sensors for detecting the presence of  $\text{H}_2\text{S}$  are known.

30           In the preferred embodiment where the  $\text{H}_2\text{S}$  measurement means is an electrochemical gas analyser or spectrophotometric analyser, the analyser may be programmed to detect when the rate of  $\text{H}_2\text{S}$  evolution has decreased.

Preferably the electrochemical gas analyser can measure the

H<sub>2</sub>S concentration on a real time basis to allow the rate of H<sub>2</sub>S evolution to be constantly monitored. Typically, the H<sub>2</sub>S evolution data may be represented as a function of cumulative H<sub>2</sub>S concentration v time or absolute H<sub>2</sub>S concentration v. time.

5 Different forms of reduced inorganic sulfur react at different rates. Thus, by being able to monitor the rate of H<sub>2</sub>S evolution, information can be obtained as to the relative amounts of different forms of reduced sulfur materials present in the sample.

10 According to a further broad form of the invention there is provided an apparatus for measuring the amount of reduced inorganic sulfur in a sample, the apparatus having a reaction chamber, a means of introducing a reducing agent that can selectively convert the inorganic reduced sulfur of the sample in the reaction chamber to hydrogen sulfide and means for continually monitoring the amount of hydrogen sulfide evolved.

15 According to still a further broad form of the invention there is provided a method of measuring the amount of reduced inorganic sulfur in a sample, the method including reacting a sample with a reducing agent that selectively converts the reduced inorganic sulfur to hydrogen sulfide and measuring hydrogen sulfide evolved as a function of time.

20 The ability to determine the relative amounts of different types of reduced inorganic sulfur is important for environmental management. This enables predictions to be made as to the potential rate of acid generation as opposed to simply calculating the total acid generating potential of a soil material.

25 A knowledge of a form of sulfur generally referred to as acid volatile inorganic sulfur is of particular importance. Acid volatile sulfur includes monosulfides and non-stoichiometric sulphides such as greigite and mackinawite. In the preferred apparatus in which acidic chromous chloride is generated *in situ*, the sample can be treated with the concentrated HCl and  
30 ethanol only. Neither chromium nor water are added.

Only the acid volatile sulfur fraction will react under these conditions and can thus be selectively analysed. If the total reduced inorganic

sulfur is required, the same sample can then be treated with the chromous chloride reductant by introducing chromium powder and water into the reaction chamber as described above.

#### BRIEF DESCRIPTION OF THE DRAWINGS

5                   Figure 1 schematically illustrates a preferred apparatus of the present invention, and

                  Figure 2 illustrates a further preferred apparatus of the present invention.

#### BEST MODE

10                  The apparatus of both Figures 1 and 2 are designed to be fully automated and to be portable to allow onsite use in the field.

                  The apparatus 11 illustrated in Figure 1 has a removable reaction vessel 12. An operator can place a known volume or mass of a sample to be tested into the reaction vessel. Alternatively, the apparatus can  
15                  include an in-built balance to measure the mass of material in the reaction chamber. The reaction vessel 12 is placed in a heating mantle 13 which is equipped with a magnetic stirrer. In use, the reaction vessel is connected, with an air-tight seal, to inlets 14, 15 and outlet 16. The sealed connection may be provided by any suitable means such as ground glass joints or O ring  
20                  seals.

                  The inlet 14 is connected to a chromium powder dispenser 17. The inlet 15 is connected, with an air-tight seal, to a nitrogen gas source 18 and to acid and water and ethanol reservoirs 19, 20. The reservoirs 19, 20, 21 each have a peristaltic pump 22, 23, 24 for delivering pre-determined  
25                  amounts of liquids via three way valves to inlet 15. The outlet 16 is connected an insulated condenser 26 which typically has copper pipes surrounding a glass core. Refrigerant is supplied to the condenser 26 from pump 27. Typically, the apparatus includes a heat exchange apparatus by which heat extracted from the condenser can be used to heat the mantle 13. The upper  
30                  end of the condenser 26 is connected, via an air-tight seal, to an IR gas analyser 28. A moisture control unit 29 is disposed between condenser and analyser 28.

The apparatus is operated by a central control computer 30. The power source 31 is typically a 12V power supply. However, mains supply of any voltage (e.g. 110V or 240V), AC or DC, may be used by the incorporation of a suitable adaptor.

5 To operate the apparatus, an operator places an amount of a sample to be tested in the reaction chamber and places the chamber in the mantle. Prior to analysis, the reservoirs of the chamber are charged with chromium powder, ethanol, water and concentrated HCl respectively. The sample to be tested may be any material containing reduced inorganic sulfur and includes soil, mine spoil, fossil fuels such as coal and oil, sediments, 10 plants and animal materials, water (both naturally occurring and waste), chemical waste and minerals. The samples may be pretreated depending on the nature of the sample. For example the mineral samples may be fine ground prior to analysis.

15 The apparatus is operated by the computer 30 which can be programmed to fully automate the analysis. If desired manual override capabilities may be included. Under normal operational conditions, all that is required of the operator after placement of the sample in the reaction chamber is to press an "on" switch. This will initiate purging of the system 20 with nitrogen for a pre-determined and monitored flow rate. After a fixed time, pre-determined amounts of chromium powder, water, ethanol and 12N HCl are added to the reaction chamber in turn. The water also serves to flush the HCl from the lines.

The amounts of reagent added will depend on the weight of the 25 sample. For a 1g sample, typically about 2.059g chromium powder, 10mL ethanol, 28.25mL of 12N HCl and 31.75mL of water are added.

After addition of the reagents, the reaction chamber is heated to boiling with stirring. As  $H_2S$  is evolved it is carried by the nitrogen through the dessicant in the moisture trap 29 to the IR gas analyser 28. The cumulative 30 concentration of the  $H_2S$  gas is calculated by the computer 30 using an algorithm relating  $H_2S$  concentration and gas flow rate. When  $H_2S$  evolution has ceased or fallen below a pre-determined level the computer will terminate

the analysis by sequentially disengaging the electrochemical gas analyser, and turning off the heating mantle 13, gas flow and condenser 26. The reaction vessel 12 can then be removed by the operator and cleaned. The apparatus is ready for the next analysis.

5           The total amount of reduced inorganic sulfur can be read directly from a digital panel or stored digitally. The results may also be presented graphically as either cumulative H<sub>2</sub>S production or real time H<sub>2</sub>S production rates.

10           The apparatus of Figure 2 is similar to that illustrated in Figure 1 and the same reference numerals refer to the same features.

15           The apparatus does not include an ethanol reservoir. Ethanol is instead added to reaction vessel 12 together with a sample for analysis. The nitrogen inlet of Figure 1 has been replaced by a pump 35 to introduce air as the carrier gas. The condensor 26 is cooled by water pumped from refrigeration unit 36 via pump 27. The IR analyser has been replaced by an electrochemical gas analyser 37. A H<sub>2</sub>S trap 38 is located between the analyser and vent 39.

20           The above procedures may be modified to separately analyse the acid volatile sulfur fraction. In this case, the sample and ethanol are placed in the reaction chamber as before, but only HCl (20mL) is added. The heating mantle is not required and remains turned off. If both the acid volatile and total reduced sulfur are required, the first procedure may then be performed on the sample remaining in the reaction chamber. However, it will only be necessary to add 8.25mL of HCl (because 20mL have already been  
25           added) plus the chromium powder and water.

30           It can be seen that the apparatus of the present invention can provide a self contained, portable and automated analyser for reduced inorganic sulfur. At present there is no automated system for quantifying reduced inorganic sulfur. All existing techniques used by industry must be performed by skilled personnel in a well equipped laboratory. Thus, it has hitherto not been possible to analyse reduced inorganic sulfur on site. Automation of the present apparatus allows onsite analysis. Further, skilled

laboratory personnel are not required to conduct the analysis.

The present apparatus can be in the form of a completely portable unit which may be operated from a 12V power supply. The present apparatus can also be operated using a mains power supply in the laboratory.

- 5 The use of recirculated chilled water contributes to the ability of the apparatus to be self contained. Such portability and self containment is not practical with conventional wet chemistry techniques.

10 The ability to constantly monitor  $H_2S$  evolution and to detect when such evolution ceases also offers significant advantages over wet chemical analytical techniques in which a sample is digested for a set period of time. The present inventors have observed that for some samples, digestion can be completed in about 10 minutes as compared to the standard time of one hour allowed for reaction. Thus by monitoring completion of the reaction, the speed of analysis can be considerably increased.

- 15 The present invention also provides a method and apparatus for selectively measuring the reduced inorganic sulfur without interference from organic sulfur and sulfate materials. Still further, the acid volatile sulfur fraction can be selectively measured. Also, by monitoring the rate of hydrogen sulfide evolution, important information can be obtained as to the relative  
20 amounts of different types of reduced inorganic sulfur in the sample.

It will be appreciated that various changes and modifications may be made to the embodiments as described and claimed herein without departing from the spirit and scope of the invention.

## CLAIMS

1. An apparatus for measuring the reduced inorganic sulfur content of a sample, the apparatus having a reaction chamber, means for introducing a reducing agent that can selectively convert the reduced inorganic sulfur of a sample in the reaction chamber to hydrogen sulfide, means for measuring the amount of hydrogen sulfide evolved by reaction of the reducing agent with the sample and a detector for detecting when evolution of the hydrogen sulfide has reduced or ceased.
2. The apparatus of claim 1, which includes a reservoir for storing a reducing agent or a precursor thereof and the reducing agent is selected from the group consisting of Cr(II), Sn(II) and Hg(II)
3. The apparatus of claim 2, wherein the reducing agent is chromous chloride.
4. The apparatus of claim 3, which includes means for introducing predetermined amounts of chromium powder and hydrochloric acid into the reaction chamber such that chromous chloride can be generated in the reaction chamber.
5. The apparatus of claim 1, which further includes a source of hydrochloric acid and means for introducing the acid into the reaction chamber prior to introduction of the reducing agent such that the amount of any acid volatile sulfur in the sample can also be measured.
6. The apparatus of claim 1 which includes a source of a carrier gas for introduction into the reaction chamber and the carrier gas can carry any evolved hydrogen sulfide to the measurement means.
7. The apparatus of claim 6, wherein the carrier gas is air and the apparatus includes a pump for pumping air into the reaction chamber.
8. The apparatus of claim 6, wherein the carrier gas is an inert gas and the apparatus includes a supply of the inert gas.
9. The apparatus of claim 1, which includes means for heating the reaction chamber.
10. The apparatus of claim 9, which includes a condenser fluidly connected to the reaction chamber and a source of refrigerated fluid for



cooling the condenser.

11. The apparatus of claim 10, which includes means for deactivating the heating means and source of refrigerated fluid when evolution of hydrogen sulfide has ceased.

5 12. The apparatus of claim 1 which includes a central control unit for controlling the introduction of the reducing agent and any other reagents to the reaction chamber.

13. The apparatus of claim 10, wherein the control unit includes means for calculating the amount of reduced inorganic sulfur in the sample  
10 from the measured sulfur hydroxide.

14. The apparatus of claim 1, which includes a means for signalling when evolution of hydrogen sulfide has ceased.

15. The apparatus of claim 1 wherein the measuring means detects when hydrogen sulfide evolution has ceased.

15 16. The apparatus of claim 14 wherein the means for measuring evolved hydrogen sulfide is selected from the group consisting of an IR spectrometer, a UV spectrometer, a gas chromatograph, and an electrochemical gas analyzer.

17. The apparatus of claim 16, wherein the measurement means  
20 continuously measures the hydrogen sulfide evolved.

18. The apparatus of claim 1 which further includes means for oxidising the hydrogen sulfide evolved and measuring the amount of oxidised hydrogen sulfide.

19. The apparatus of claim 1, wherein the sample is selected from  
25 the group consisting of sediment, soil, sludge, petroleum, mine spoil, coal, oil, water, plant, animal and mineral material.

20. The apparatus of claim 19, wherein the sample includes organic sulfur and/or a mineral sulfate.

21. The apparatus of claim 19, which is in the form of a portable unit.

30 22. A method of measuring the amount of reduced inorganic sulfur in a sample, the method including reacting a sample with a reducing agent that selectively converts the reduced inorganic sulfur to hydrogen sulfide and

measuring the hydrogen sulfide evolved as a function of time.

23. The method of claim 22, wherein the reducing agent is selected from the group consisting of Cr(II), Sn(II) and Hg(II).

24. The method of claim 23, wherein the reducing agent is  
5 chromous chloride.

25. The method of claim 22, wherein means for securing the hydrogen sulfide evolved is selected from an IR spectrometer, a UV spectrometer, a gas chromatograph, and an electrochemical gas analyzer.

26. The method of claim 22, wherein the sample is selected from the  
10 group consisting of sediment, soil, sludge, petroleum, mine spoil, coal, oil, water, plant, animal or mineral material.

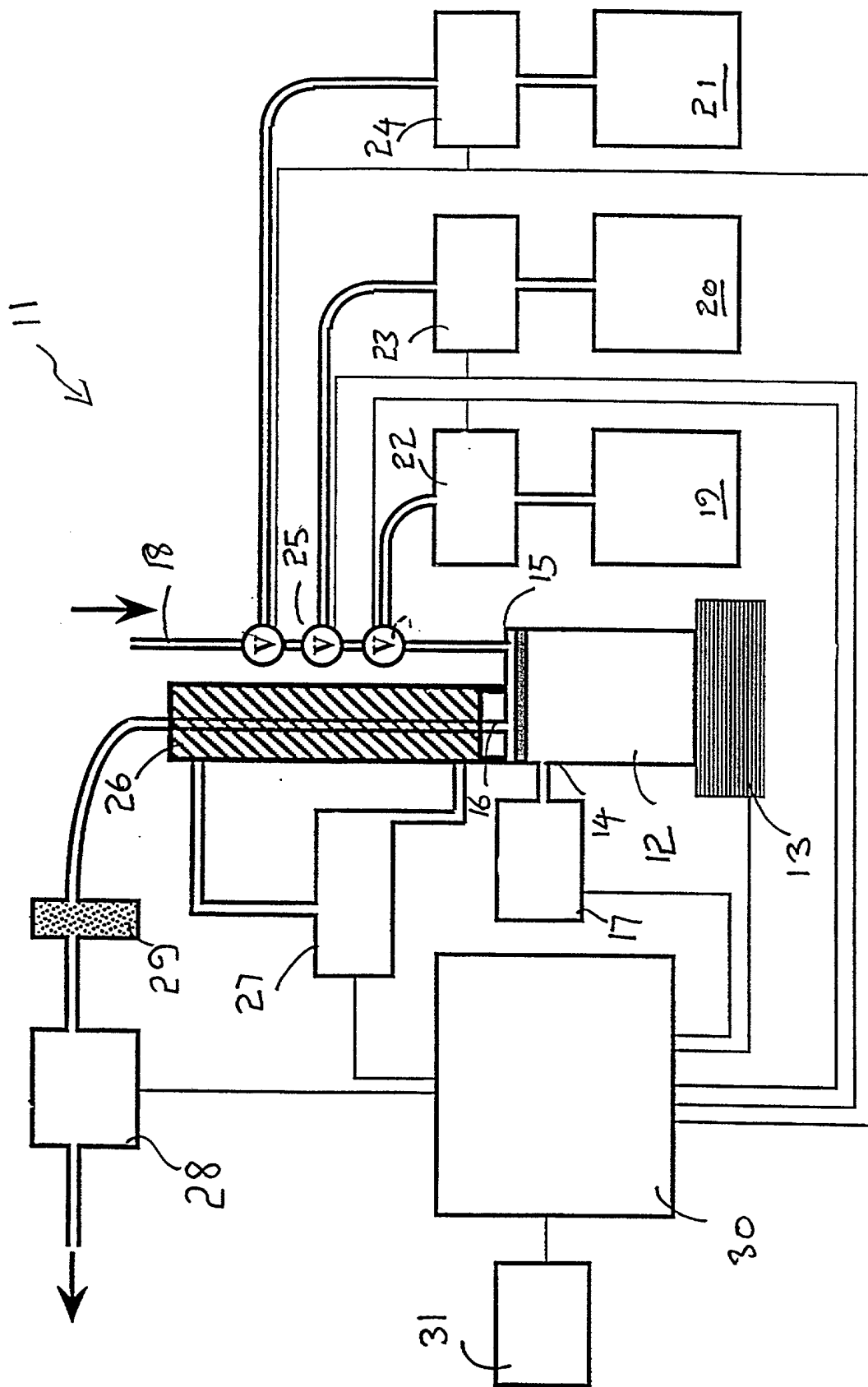


Fig. 1

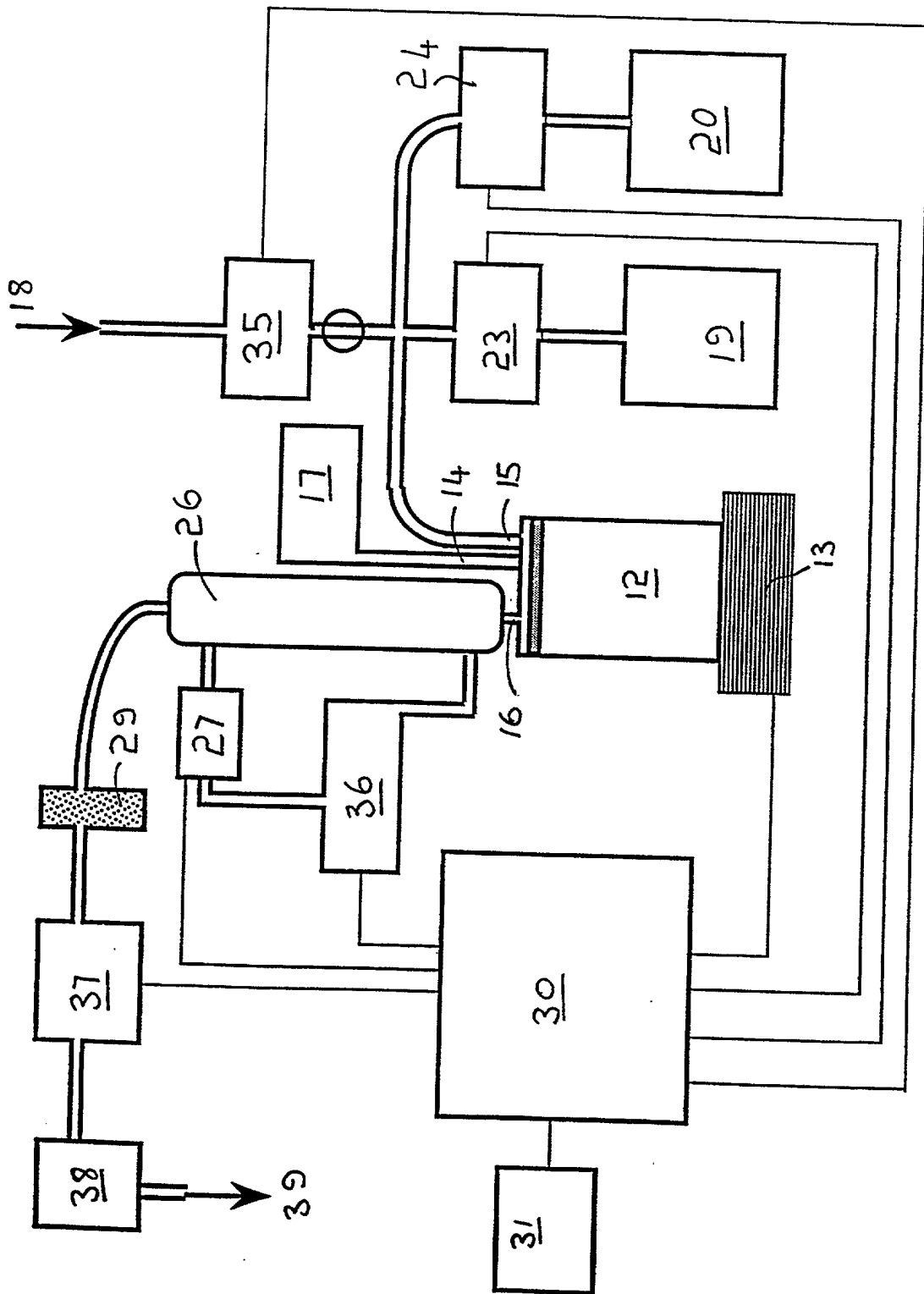


Fig. 2

Attorney's Docket No. \_\_\_\_\_

**COMBINED DECLARATION AND POWER OF ATTORNEY***(ORIGINAL, DESIGN, NATIONAL STAGE OF PCT OR CIP APPLICATION)*

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name, I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

Apparatus for Analysing Reduced Inorganic Sulphurthe specification of which: *(complete (a), (b) or (c) for type of application)***REGULAR OR DESIGN APPLICATION**

- (a) ☐ is attached hereto.  
(b) ☐ was filed on \_\_\_\_\_ as Application Serial No. \_\_\_\_\_ and was amended on \_\_\_\_\_ *(if applicable)*.

**PCT FILED APPLICATION ENTERING NATIONAL STAGE**

- (c) ☒ was described and claimed in International Application No. PCT/AU00/00024 on 22 March 2000 and as amended on \_\_\_\_\_ *(if any)*.

**ACKNOWLEDGEMENT OF REVIEW OF PAPERS AND DUTY OF CANDOR**

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations. § 1.56(a).

- ☒ In compliance with this duty there is attached an information disclosure statement. 37 CFR 1.97.

**PRIORITY CLAIM**

I hereby claim foreign priority benefits under Title 35, United States Code, § 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed.

RENNER, OTTO, BOISSELLE & SKLAR  
(Declaration and Power of Attorney—page 1 of 3)

(complete (d) or (e))

- (d) ☐ no such applications have been filed.  
 (e) ☒ such applications have been filed as follows

EARLIEST FOREIGN APPLICATION(S), IF ANY FILED WITHIN 12 MONTHS  
 (6 MONTHS FOR DESIGN) PRIOR TO SAID APPLICATION

Country	Application No.	Date of filing (day, month, year)	Date of issue (day, month, year)	Priority Claimed
Australia	PP9347	22.03.1999		<input checked="" type="checkbox"/> YES NO <input type="checkbox"/>
				<input type="checkbox"/> YES NO <input type="checkbox"/>
				<input type="checkbox"/> YES NO <input type="checkbox"/>

ALL FOREIGN APPLICATION(S), IF ANY FILED MORE THAN 12 MONTHS  
 (6 MONTHS FOR DESIGN) PRIOR TO SAID APPLICATION

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CONTINUATION-IN-PART

(complete this part only if this is a continuation-in-part application)

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, § 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
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(Application Serial No.)	(Filing Date)	(Status) (patented, pending, abandoned)
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POWER OF ATTORNEY

As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (List name and registration number)

Armand P. Boisselle  
 Reg. No. 22,381

Don W. Bulson  
 Reg. No. 28,192

Warren A. Sklar  
 Reg. No. 26,373

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RENNER, OTTO, BOISSELLE & SKLAR  
 (Declaration and Power of Attorney—page 2 of 3)

## SEND CORRESPONDENCE TO

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 RENNER, OTTO, BOISSELLE & SKLAR  
 1621 Euclid Avenue  
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DIRECT TELEPHONE CALLS TO:  
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Don W. Bulson  
 (216) 621-1113

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first inventor SULLIVAN, Leigh Albert

Inventor's signature Leigh Sullivan

Date 10 Sep 2001 Country of Citizenship AU

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Full name of second joint inventor, if any MCCONCHIE, David Murray

Inventor's signature David Murray

Date 10 Sep 2001 Country of Citizenship AU

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- ☒ Signature for third and subsequent joint inventors. Number of pages added 1
- ☐ Signature by administrator(trix), executor(trix) or legal representative for deceased or incapacitated inventor. Number of pages added \_\_\_\_\_
- ☐ Signature for inventor who refuses to sign or cannot be reached by person authorized under 37 CFR 1.47. Number of pages added \_\_\_\_\_

Please type a plus sign (+) inside this box → ☐

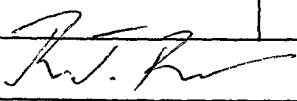
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**DECLARATION****ADDITIONAL INVENTOR(S)**  
**Supplemental Sheet**  
Page 1 of 1

Name of Additional Joint Inventor, if any:		<input type="checkbox"/> A petition has been filed for this unsigned inventor					
Given Name (first and middle [if any])				Family Name or Surname			
300 Richard				BUSH			
Inventor's Signature						Date	12/09/2001
Residence: City	GOONELLABAH	State	NSW	Country	AUSTRALIA	Citizenship	AU
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Office Address							
City	GOONELLABAH	State	NSW	ZIP	2480	Country	AUSTRALIA
Name of Additional Joint Inventor, if any:		<input type="checkbox"/> A petition has been filed for this unsigned inventor					
Given Name (first and middle [if any])				Family Name or Surname			
Inventor's Signature						Date	
Residence: City		State		Country		Citizenship	
Post Office Address							
Post Office Address							
City		State		ZIP		Country	
Name of Additional Joint Inventor, if any:		<input type="checkbox"/> A petition has been filed for this unsigned inventor					
Given Name (first and middle [if any])				Family Name or Surname			
Inventor's Signature						Date	
Residence: City		State		Country		Citizenship	
Post Office Address							
Post Office Address							
City		State		ZIP		Country	

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(July 1998)